



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2581

Powdered Paint
Nominal 0.5 % Lead

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and for the calibration of apparatus used to determine lead in paint. SRM 2581 is composed of paint collected from the interior surfaces of housing. A unit consists of 35 g of powdered paint material, 99+ % of which passes a 100 μm (No. 145) sieve. The certified mass fraction of lead, given below, is based on measurements by isotope dilution inductively coupled plasma mass spectrometry (ID-ICPMS) with a minimum sample size of 100 mg. The certified value is reported on a dry basis (see Instructions for Drying).

Certified Mass Fraction

Lead Content: 0.449 % \pm 0.011 %

The uncertainty in the certified value is calculated as

$$U = k u_c$$

where u_c is the combined standard uncertainty calculated according to the ISO Guide [1] and k is a coverage factor. The value of u_c is intended to represent at the level of one standard deviation, the combined effect of uncertainty components associated with material inhomogeneity and ID-ICPMS measurement uncertainty. In the absence of Type B uncertainties (which are negligible here in comparison with Type A), the expanded uncertainty (U) given is for a 95 % prediction interval. The coverage factor, $k = 2.57$, is the Student's t -value for a 95 % prediction interval with 5 degrees of freedom.

NOTICE AND WARNING TO USERS

Expiration of Certification: The certification of this SRM lot is valid within the measurement uncertainties specified until December 31, 2010, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see Use). However, the certification will be nullified if the SRM is contaminated or modified.

Stability: This material is considered to be stable. NIST will monitor this material and will report any substantial changes in certification to the purchaser. Return of the attached registration card will facilitate notification.

Use: To relate analytical determinations to the certified value on this Certificate of Analysis, a minimum sample mass of 100 mg should be used and the sample should be dried according to the Instructions for Drying. Sample preparation procedures should also be designed to effect complete dissolution in order to relate the determined value to the certified value. This SRM must be stored in an air conditioned environment or similar cool and dry environment away from sunlight and fumes.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by B.S. MacDonald.

The overall direction and coordination of the technical measurements leading to certification of this SRM were performed by J.R. DeVoe, P.A. Pella, and R.L. Watters, Jr. of the NIST Analytical Chemistry Division.

Statistical consultation was provided by S.D. Leigh of the NIST Statistical Engineering Division.

Partial financial support for the development of this SRM was provided by the U.S. Environmental Protection Agency (EPA) under the direction of project managers S.L. Harper and M.E. Beard of the EPA Office of Research and Development, National Exposure Research Laboratory, Research Triangle Park, NC.

COLLECTION, PREPARATION, AND ANALYSIS

Collection: The paint material for this SRM was collected primarily in North Carolina from various interior wall surfaces of old housing which, for the most part, were painted prior to 1945. The material was collected under the direction of the Research Triangle Institute and the U.S. Environmental Protection Agency. Collection of paint by dry scraping and its initial evaluation for use as SRM 2581 were performed by J.D. Neefus, E.E. Williams, and D.B. Binstock, of the Research Triangle Institute, Research Triangle Park, NC, under the leadership of W.F. Gunknecht.

Preparation: The largest pieces of debris and foreign material were first removed by hand. Next, the material was coarsely chipped in a large-capacity blender fitted with a stainless steel blade. The material was then further ground in small batches in a ball mill. Each batch was sieved and the fraction that did not pass a 100 μm (#145) sieve was returned for further grinding with a fresh charge of coarse paint material. All material of a size less than 100 μm was combined and blended as a single batch before being bottled in 35 g units.

Analysis: Certification analysis by ID-ICPMS was performed by E.S. Beary and K.E. Murphy of the NIST Analytical Chemistry Division. X-ray fluorescence homogeneity analysis was performed by A.F. Marlow and P.A. Pella and inductively coupled plasma-optical emission spectrometric (ICP-OES) analysis was performed by L.J. Wood of the NIST Analytical Chemistry Division.

The ICP-OES analysis data given in Table 1 provide information on the concentrations of major constituents other than lead in the material. These values listed are not certified, but are given only to provide additional information on the matrix.

Instructions for Drying: Samples of this SRM should be dried in an air atmosphere at 105 °C for 2 h. At NIST, loss on drying according to this procedure was about 1 % relative by mass. However, under different conditions of humidity, the mass loss could vary. In order for users to directly relate their analyses to the certified value, loss on drying corrections should be measured and applied at the time of the analysis.

Environmental Lead Proficiency Analytical Testing Program Results: This material was included as an unknown in the Environmental Lead Proficiency Analytical Testing Program (ELPAT) administered by the American Industrial Hygiene Association (AIHA). Conventional dissolution methods employed by participating laboratories include hotplate, microwave, and other techniques such as sealed bomb dissolutions and leaching techniques. Instrumental determinations were performed using inductively coupled plasma mass spectrometry (ICPMS), inductively coupled plasma-optical emission spectrometry (ICP-OES), flame atomic absorption spectrometry (FAAS), graphite furnace atomic absorption spectrometry (GFAAS) and X-ray fluorescence spectrometry (XRF). Information from this study is provided to indicate the state of the practice for lead in paint measurements using such methods. A summary of the round robin lead results obtained from ELPAT Reference Laboratories for SRM 2581 is presented in Table 2. The SRM 2581 is identified as round robin 015, paint #3 in the ELPAT report.

SUPPLEMENTAL INFORMATION

Table 1. Information Values for Major Constituents of SRM 2581

Element	Mass Fraction (%)
Al	2
Ca	11
Fe	0.4
Mg	1
Ti	12
Zn	2

Table 2. Environmental Lead Proficiency Analytical Testing (ELPAT) Program
Summary Statistics of Reference Laboratories for Round Robin 015*

Sample	n	Mean	Minimum	Maximum	s ^b		
Paint 3	80	0.417 %	0.349 %	0.470 %	0.030		

*These results are provided to demonstrate user experience with this material. They were not used in calculating the certified value of SRM 2581.

^bs is one standard deviation.

REFERENCE

- [1] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993). See also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington, D.C., (1994).